Effects of Specimen Size on Bending Strength and Fracture Toughness of AlN

Chang-Sam Kim, Toyohiko Yano*, and Takayoshi Iseki

Department of Inorganic Materials, Tokyo Institute of Technology, O-okayama, Meguro-ku, Tokyo 152, Japan
* Research Laboratory for Nuclear Reactors, Tokyo Institute of Technology, O-okayama, Meguro-ku, Tokyo 152, Japan

ABSTRACT

Bending Strength and fracture toughness of pressureless-sintered AlN were measured at room temperature by three-point bending test and SEPB (Single Edge Precracked Beam) method, respectively. Four batches of different-sized specimens were used for the bending test. It was found that a degree of strength discrepancy between the measured and an ideal strength obtained from a size-effect relation due to Weibull had a tendency to be increased with decreasing the specimen size. A major reason of this increment was caused by an effect of different probability of fracture from surface among the batches.

Fracture toughness values obtained from 2 mm × 2 mm × 15 mm, 2 mm × 4 mm × 25 mm and 3 mm × 4 mm × 25 mm specimens were 2.5, 2.7 and 2.7 $MPa \cdot m^{1/2}$, respectively. The standard deviation in K_{Ic} of each batch was < 0.20 $MPa \cdot m^{1/2}$.

INTRODUCTION

AlN is recognized as a potential material for nuclear reactor devices, e.g. direct converter insulator and magnetic coil insulator, because of its useful thermal and electrical properties[1,2]. Its thermal conductivity (~200 $W/(m \cdot K)$) is nearly equal to that of BeO. AlN also has an excellent electrical resistivity and a high fracture strength. A prime requirement in this application is retention of adequate strength after neutron irradiation, to resist structural, thermal or swelling stresses.

There have been attempts to reduce the specimen size for mechanical testing, especially for the testing of irradiated specimens, but most of the attempts were concerned in metal or alloy [3,4,5], while a few in ceramics[6,7]. Major motivations to establish small specimen testing methods are limitations in irradiation space and radiation dose to personnel in post-irradiation tests.

The first stage of the attempts is to obtain basic data for unirradiated specimens, such as effect of specimen size and/or shape, surface finishing and strain rate, for ceramics. The basic data for Al_2O_3 and SiC[6] have been reported, but not yet for AlN despite of its potential.

This paper reports effect of specimen size on strength and fracture toughness of AlN by comparing the results of the different-sized specimens.

EXPERIMENTAL PROCEDURE

Pressureless-sintered AlN† was used in this work. Its purity and density were >99.5% and 3.25 g/cm^3, respectively. The edges of the test pieces were manually chamfered with a diamond grinding disc, of which mean grit is 15 μm. The surfaces were wet polished with alumina powder; the maximum surface roughness was 0.5 μm. Dimensions of the specimens for bending test were nominally 0.8 mm × 2 mm × 15 mm, 2 mm × 2 mm × 25 mm, 2 mm × 4 mm × 25 mm and 3 mm × 4 mm × 40 mm, and these for fracture toughness test were nominally 2 mm × 2 mm × 15 mm, 2 mm × 4 mm × 25 mm and 3 mm × 4 mm × 25 mm.

Bending strength was measured by three-point bending test at room temperature. Strain rate was maintained to be constant for all the specimens by 1.7 × 10^{-5}sec^{-1}. A commercial bending jig made of hardened steel was used for the test in which support span length was longer than 20 mm. While a miniature bending jig was used for the testing of 8 mm in the support span length. Schematic of the miniature jig is shown in Fig. 1.

Fracture toughness testing was proceeded according to JIS R 1607 (Testing Methods for Fracture Toughness of High Performance Ceramics). At first, Vickers indentation was placed as a precrack starter at the center of tensile surface with an indentation load from 1 kg to 10 kg. The specimen with a precrack starter was set upon a bridging jig‡ of its groove width 3 mm, and loaded manually until a pop-in sound was detected; the compressive load was then decreased immediately. This specimen was three-point bending tested at room temperature. The support span was 4 times the width of specimen, and cross head speed was 0.01 mm/min. The precrack was inspected after the bending test and it was determined whether the precrack would meet the

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† Shapal SH-15, Tokuyama Soda Co., Tokyo, Japan.
‡ MBK-603C, Maruto Instrument Co., Tokyo, Japan.

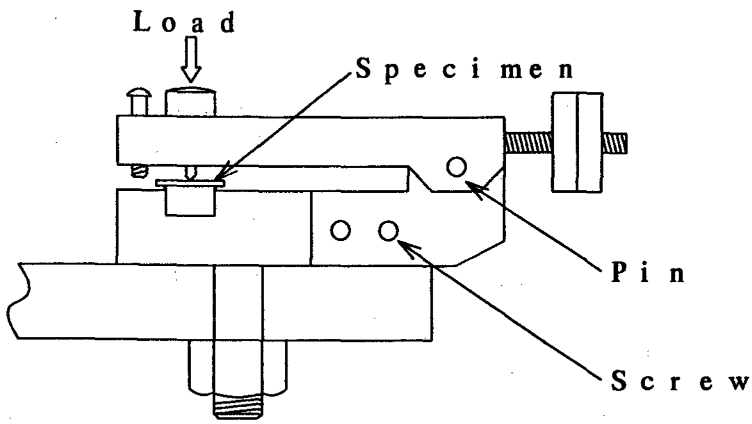

Fig. 1 Schematic of three-point bending jig for miniature specimen.

requirements of JIS R 1607; K_{Ic} was then calculated.

RESULTS AND DISCUSSION

Bending Strength

The bending strength and Weibull modulus obtained by the maximum likelihood method are summarized in Table 1. The bending strength of 2 mm × 2 mm × 25 mm specimens as well as 2 mm × 4 mm × 25 mm specimens is lower than that of 3 mm × 4 mm × 40 mm specimens despite of an effective volume of the former specimens is smaller than that of the latter ones. These results are not compatible to a general trend of change in measured strength based on the size-effect relation due to Weibull. By Weibull's analysis, average strengths, σ_1 and σ_2, of two different sizes or shapes of components made in the same way with the same material and stressed in the same way are in a ratio given by:

$$\sigma_1/\sigma_2 = (V_{e2}/V_{e1})^{1/m} \quad (1)$$

where V_{e1} and V_{e2} are the effective volume of component 1 and 2, respectively: m is the Weibull modulus. The effective volumes of 0.8 mm × 2 mm × 15 mm specimens and 3 mm × 4 mm × 40 mm specimens are 0.053 and 1.488, respectively, and σ_1/σ_2 is 1.40 in the case that m is 10. Considering this ratio of strengths, the measured strength of 0.8 mm × 2 mm × 15 mm specimens is too low to rationalize the difference between the measured strengths of these specimens with the equation (1).

Degree of strength discrepancy (DSD) between the measured strength, σ_m, and the ideal fracture stress, σ_i, calculated from the equation (1) for a batch of specimens is defined as:

$$DSD = \frac{(\sigma_i - \sigma_m)}{\sigma_i} \times 100 \qquad (2)$$

DSD of AlN specimens has a tendency to increase with decreasing the specimen size, and the same tendency has been reported for Al_2O_3 and SiC specimens thinner than 3 mm in thickness[6]. Some reasons of the increment in DSD have been considered, such as effects of surface finishing and strain rate[7]. However it was found that these effects hardly affected the measured strength. It was proposed that these increments would be attributed to an effect of higher probability of fracture from surface for the smaller specimens, because flaws located at the surface has a higher stress intensity factor than flaws in the bulk. It has been confirmed for various-sized SiC specimens that a proportion of fracture from the surface of a batch increased as the specimen size decreased[6]. Therefore, it is satisfactory to consider the effect of different proportion of fracture from the surface as a principal reason of increment in DSD for AlN.

These results of the present and the cited works suggest that it is not unusual that the fracture strength of smaller specimen is less than that of larger one for ceramic specimens thinner than 3 mm in thickness.

Fracture Toughness

Measured fracture toughness is summarized in Table 2. The number of specimens with a proper precrack satisfying the requirements of JIS R 1607, is also summarized in Table 2. The fracture toughnesses of 2 mm × 4 mm × 25 mm and 3 mm × 4 mm × 25 mm specimens are the same value, while that of 2 mm × 2 mm × 15 mm specimens is slightly lower by 8% than the others. However, it was difficult to introduce the proper precrack as the specimens became smaller. The relations between the fracture toughness and a ratio of the precrack-length to specimen width (a/w) are shown in Fig. 2. The fracture toughness increased ~20% with increasing a/w from 0.26 to 0.47. This increase might be caused by a stable crack growth after the pop-in crack formation for the specimens of higher a/w. The precrack length is varied by changing the indentation load and/or the groove width of the

Table 1 Result of Bending Strength Measurements

Dimension* (mm)	Span length (mm)	Number of specimens	Bending strength (MPa)	Weibull modulus
3 × 4	30	15	323 ± 41	9
2 × 4	20	15	314 ± 25	12
2 × 2	20	20	314 ± 28	12
0.8 × 2	8	30	342 ± 43	8

* thickness × width

Table 2 Result of Fracture Toughness Measurements

Dimension* (mm)	Number of specimens	Number of desirable specimens§	Fracture toughness ($MPa \cdot m^{-1/2}$)
3 × 4	10	10	2.7 ± 0.20
2 × 4	10	7	2.7 ± 0.14
2 × 2	15	9	2.5 ± 0.16

* thickness × width

§ specimens with a proper precrack satisfying the requirements of JIS R 1607

bridge jig, generally the lower the indentation load and/or the wider the groove width introduce the longer the precrack length[8]. In the present work, specimens of lower indentation load, which need relatively higher compressive load, had been also a relatively higher a/w. Hence, there would be much chance to propagate a stable crack for the specimens with higher a/w than those with lower a/w.

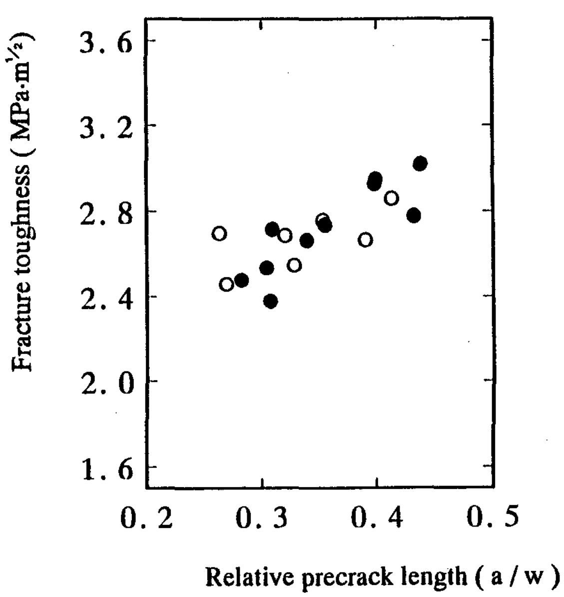

Fig. 2 Values of fracture toughness vs relative precrack length (a/w): ● 3 mm × 4 mm × 25 mm, ○ 2 mm × 4 mm × 25 mm.

SUMMARY

Bending strength of the specimens thinner than 3 mm in thickness was lower than the ideal strength predicted from the size-effect relation due to Weibull for AlN tested. A major reason of the discrepancy in these strengths might be the different probability of fracture from the surface between batches.

Fracture toughness measured by SEPB method for 2 mm × 2 mm × 15 mm specimens was nearly equal to the result of 3 mm × 4 mm × 25 mm specimens, but it was difficult to introduce a proper precrack satisfying the requirements of JIS R 1607 in the case of the smaller specimens.

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REFERENCES

1) R. Heidinger and F. Königer, "Investigations on Advanced Ceramics for Window Applications in Radiofrequency Heating Concepts," J. Nucl. Mater., **155-157**, 344-47 (1988).
2) T. Noda, F. Abe, H. Araki, and M. Okada, "Materials Selection for Reduced Activation of Fusion Reactors," J. Nucl. Mater., **155-157**, 581-84 (1988).
3) F. G. Nelson, P.E. Schilling, and J. G. Kaufman, "The Effect of Specimen Size on the Results of Plane-Strain Fracture-Toughness Tests," Eng. Frac. Mech., **4**, 33-50 (1972).
4) M. L. Hamilton and F. H. Huang, "Use of the Disk Bend Test to Assess Irradiation Performance of Structural Alloys," in The Use of Small-Scale Specimens for testing Irradiated Material, Edited by W.R. Corwin and G.E. Lucas, ASTM-STP 888, American Society for Testing and Materials, Philadelphia, 1986, pp. 5-16.
5) A. Kohyama, K. Hamada, and H. Matsui, "Specimen Size Effects on Tensile Properties of Neutron-Irradiated Steels," J. Nucl. Mater., **179-181**, 417-20 (1991).
6) C. S. Kim and T. Iseki, "Three-Point Bending Test Using Miniature Ceramic Specimens," J. Am. Ceram. Soc., to be submitted.
7) C. S. Kim, unpublished work.
8) T. Nose and T. Fujii, "Evaluation of Fracture Toughness for Ceramic Materials by a Single-Edge-Precracked-Beam Method," J. Am. Ceram. Soc., **71** [5] 328-33 (1988).